

# Photoluminescence Studies and Characterization of $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$ Red Emitting Phosphor

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## ABSTRACT

The PL emission and excitation spectrum of prepared  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}:\text{Eu}^{3+}$  phosphor explained in which excitation at wavelength of 396 nm and it prepared by combustion synthesis method for the emission spectrum exhibits a well defined asymmetric band with maximum emitting a red light give isolated red emission at 593 nm & 620 nm for 1 mol %, which has higher PL emission concentrations. The samples were characterized by XRD, SEM, PL and CIE color co-ordinates techniques with relation of emission intensity w.r.to. concentrations of rare earth ion. This process enables quick and energy-efficient generation of well-crystallized particles in fine powder form within a little action. The emission spectra are clearly from the same observable site. No another emission band was observed in emission spectrum indicating that trivalent europium ion occupies one category of sites in the host material

**Keywords :** XRD, SEM, PL, CIE, Luminescence

## I. INTRODUCTION

The PL emission and excitation spectrum of prepared  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}:\text{Eu}^{3+}$  phosphor shown in fig.3 and fig.4. The PL emission spectrum which has been shown in figure and excitation at wavelength of 396 nm and it prepared by combustion synthesis method. The fig 6.28 , shows excitation spectrum peaking at 396 nm with a broad spectral band.[1-2-3] On excitation at 396 nm, the emission spectrum exhibits a well defined asymmetric band with maximum at 593 nm & 620 nm emitting a red light give isolated red emission at 593 nm & 620 nm for 1 mol %, which has higher PL emission concentrations.[4-5] Because of the spectroscopic properties of  $\text{Eu}^{3+}$  with favorable

response and the ability to integrate the  $\text{Eu}^{3+}$  ion into diverse host inorganic material which was activated with europium ion cover transformed interest for most of the applications [4]. These materials recombine with high light capitulate, constructive emission wavelength, faster fluorescence decay with stability of temperature which make them attractive for useful in detectors for elevated energy physics [5] and also medical imaging [6]. Therefore, with the support of outstanding luminescence properties, inorganic materials activated with  $\text{Eu}^{3+}$  ions are applied in lightings industries, detectors[7] with display systems for ionizing radiation [8-9]. To calculate the 5d energies of another rare earth ion in same host lattices material  $\text{Eu}^{3+}$  ion also is used as a

reference ion [9]. So, the spectroscopic properties investigation of trivalent europium ion in various host material is important for both the actual applications and the fundamental research. Combustion method is not just the other synthesis process known as combustion synthesis method [10-11] which does not required further repeated heating and calcinations. It is just not other than exothermic reaction and occurs with evaluation of light and heat. For the combustion synthesis process oxidizer with fuel are required, in which oxidizing reactant viz., metal nitrates and urea work as a reducing reactant. The startup composition of the strontium nitrate, aluminum nitrate, sodium fluoride and quantity of urea was depends on the total and reducing and oxidizing valences of the fuel and oxidiser using the concept of propellant chemistry [12].

The combustion synthesis process was proceed out approximate at the temp of 550°C using Merck analytical grade inorganic materials which are as follows  $\text{Ca}(\text{NO}_3)_2$  (99.99% purity Merck),  $\text{Pb}(\text{NO}_3)_2$  (99.99% purity Merck),  $\text{KPO}_4$  (99.99% purity Merck),  $\text{NH}_4\text{Cl}$  (99.99% purity Merck), urea ( $\text{NH}_2\text{-CO-NH}_2$ ) (99.99% purity Merck) was used as fuel  $\text{Eu}(\text{NO}_3)_3$ , (REI 99.9 %). The concentration of  $\text{Eu}^{3+}$  varies as 1-0.1 mol %.

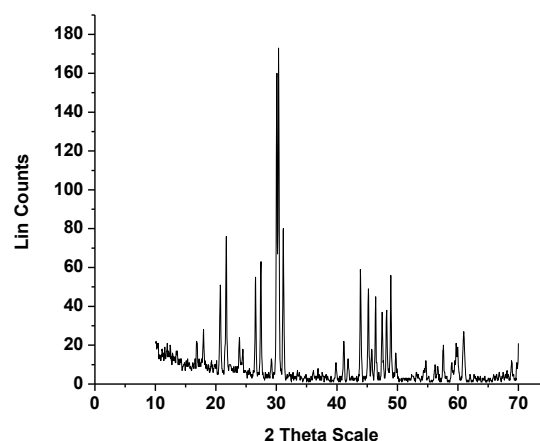
All the ingredient were assorted according to stoichiometric ratio in agate mortar and finally a pasty solution was formed, after that the solution is then shifted to silica crucible, and kept inside in a muffle furnace which was maintained at a constant temperature of about 550°C. Foamy powder with the flame was formed and that powder was collected and further analyzed for emission and excitation spectra for photoluminescence measurement.

## Results and discussion

### X-ray diffraction study

The prepared powder compound was characterized for study of its phase purity and crystallinity by X-ray powder diffraction (XRD) using a PAN Analytical X'pert Pro diffractometer XRD diffractometer for study of the XRD pattern of prepared powder

compound was recorded using  $\text{Cu-K}\alpha$  of radiation (1.54060 nm) with a scanning scan step time 10.3377s with continuous scan type. We prepared this  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}:\text{Eu}^{3+}$  materials  $\text{Eu}^{3+}$  ion on the reference to find the XRD pattern but in order to explore the crystal phase structure the pure compound was confirmed by measuring the X-ray diffraction (XRD) pattern but it is a novel compound so we maintain the data received from measuring centre for confirmation of phase and purity. The XRD pattern of the  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}:\text{Eu}^{3+}$  compound is as fallows. The XRD-patter shows phosphor have good crystalline nature. The X ray diffraction pattern of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}:\text{Eu}^{3+}$  compound is shown in following figure 1.

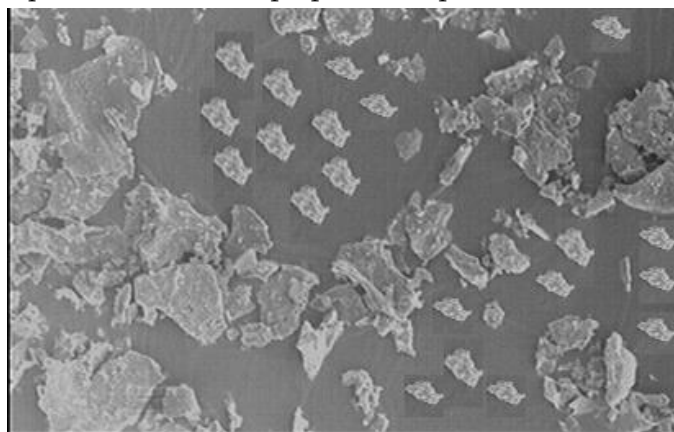


**Fig.1** XRD-pattern of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}$  phosphor.

### Scanning electron microscopic study

To investigate the surface morphology S.E.M. study was carried out and the crystallite sizes of the synthesized phosphor powder. The combustion synthesis was carried out at the temp of 550°C by heating reaction. This explains that the combustion process of the mixtures took place very well. The distinctive morphological images are represented in fig. 2, for  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}$  prepared phosphor materials correspondingly. It is clearly observed that the crystallite sizes in micrographs vary from few microns to numerous tens of microns. The crystallites contain

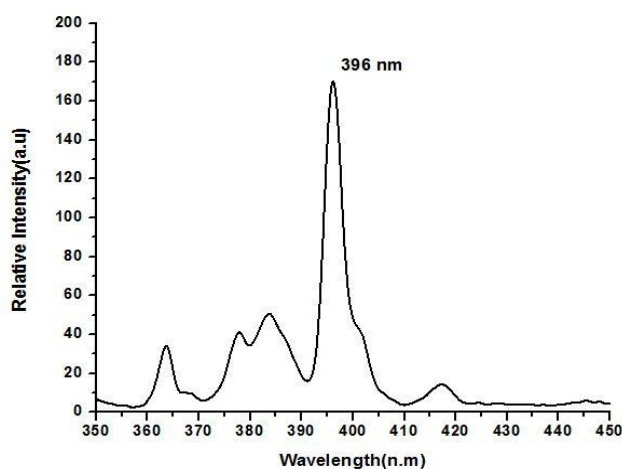
intelligent surface morphological images and contain crystalline grains. The particles possess foamy like morphology formed from highly agglomerated crystallites. The standard crystallite size is in sub-micrometer assortment as shown in SEM micrographic images; the crystallite sizes are almost equivalent for all the prepared compositions.



**Fig.2** SEM morphological images of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}$  phosphor

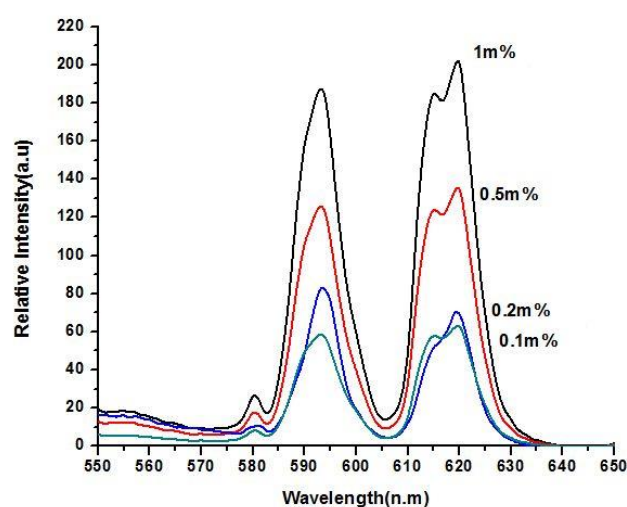
#### Photoluminescence studies of $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$ red emitting phosphor.

Fig. 3 and fig 4 shows photoluminescence excitation and emission spectra of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  phosphors which shows a wide absorption band due to the  $4f-5d$  transition of  $\text{Eu}^{3+}$  ions peaking at 396 nm. The PL emission spectra of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  phosphor as shown in fig 3 and fig 4 which exhibit red emission band observed at 593nm & 620 nm.



**Fig. 03.** Excitation spectra for  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$   
 $\lambda_{\text{em}}: 593 \text{ nm} \& 620 \text{ nm}$

The  $\text{Eu}^{3+}$  activated  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl}$  phosphor, having doping concentration varying from 1m% to 0.1m % excited by using wavelength 396 nm showing main emission peaks at 593 nm and 620 nm. As the concentration of trivalent europium ion increases, the significant virtual intensity of both transitions such as 593 ( $^5\text{D}_0 \rightarrow ^7\text{F}_1$ ) and 620 ( $^5\text{D}_0 \rightarrow ^7\text{F}_2$ ) increases. From the emission spectrum of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  phosphor, suitable for red emission for the purpose of solid state lighting. The emission spectrum is clearly from the same observable site. No another emission band was observed in emission spectrum indicating that  $\text{Eu}^{3+}$  ion occupies one category of sites in the host material.

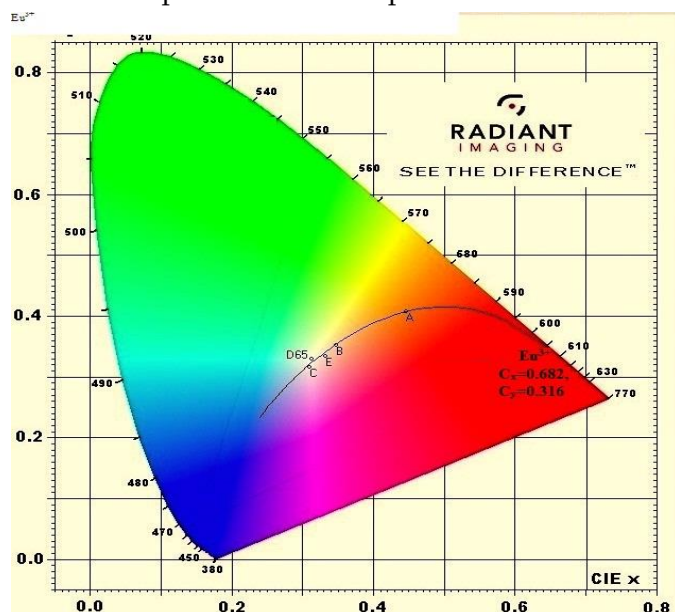


**Fig. 04** Emission spectra for  
 $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$ ,  $\lambda_{\text{ex}}: 396 \text{ nm}$ .

#### Chromatic properties of $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$ phosphor

We think that as  $\text{Eu}^{3+}$  is present with host lattice then the quantity of energy can be shifted to the activator ion, resulting from the distinctive unique emission peak of these activator ions. [13-6] It is renowned fact that the outcome obtained from the luminescence properties of prepared inorganic phosphors in powder form depends on the concentration of activator ion, therefore the recognition with concentration of dopant is necessary [14-15-16-17]. Consider the emission spectrum of  $\text{Eu}^{3+}$  which was located in red region, was chosen for further analytical study and characterization, the luminescent properties of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  red emitting phosphors and

achieve the complete emission of color. Here we determine the coordinate of chromaticity indexed with the help of the emission spectra of  $\text{Eu}^{3+}$ .



**Fig 05.** CIE chromatic diagram for  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  phosphor.

The CIE diagram of  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  red emitting phosphor shown in  $0.682 C_y = 0.316$ . With the help of CIE diagram it is easy to explain that the  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  phosphors are very near to the CIE graph frame, which easily shows the utmost color clarity of prepared phosphor material.

## II. CONCLUSION

In this research work  $\text{Ca}_2\text{Pb}_3(\text{PO}_4)_3\text{Cl} : \text{Eu}^{3+}$  red emitting lamp phosphor prepared with the help of combustion synthesis process. This process enables quick and energy-efficient generation of well-crystallized particles in fine powder form within a little action. High resolution scanning electron micrographic study shows the existence of numerous small particles inside the plates as well as regular crystalline grains in observed in the range of micron to sub-microns. The emission bands obtained at 593 nm & 620 nm which is suitable for the red emission for the lamp phosphor.

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